## Comparison of Carbon Thermal Spray Material and Carbon Nanotubes Using Carbon XANES Spectroscopy

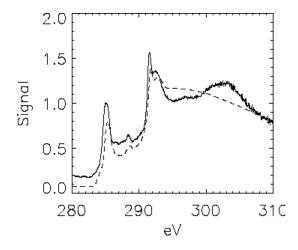
S. Wirick, R. Goswami, J. Parise, (SUNY, StonyBrook), G. Appel and H. Ade (NCSU) Abstract No. Wiri5812 Beamline(s): X1A

Microtomed samples of a thermal spray material were analyzed at the NSLS at beamline X1A at energies between 280-310 eV. The high velocity thermal spray process takes ~70 micron diameter nickel coated graphite spheres, heats these particles to 2000°C and accelerates the spheres to a maximun velocity of 700 m/sec. The particles impact a steel substrate generating pressures between 15-23 Gpa, producing nanodiamonds less than 10 nm in size (Goswami, et al.,2000). The presence of an elliptical ring around the (0002) reflection of graphite in the electron diffraction pattern suggests that the sample may also contain carbon nanotubes.

At the ALS, spectra were obtained using beamline 7.0.1.1 from carbon nanotubes made by Hideo Shimada and purified by Leslie Fleming. These nanotubes were produced by physical laser vaporization using graphite pellets which contained 0.3% Co/Ni catalyst. The sample was then refluxed in hydrogen peroxide to remove amorphous carbon, washed with carbon disulfide to remove fullerenes and filtered to remove any remaining catalyst.

Figure 1 shows a comparison of a spectrum obtained from the nanotubes and a spectrum from the carbon thermal spray sample. The peaks are slightly shifted relative to one another due to a discrepancy between the calibration of the ALS and NSLS monochromators. The spectrum from the ALS (dotted line) were collected using a procedure whereby images taken at closely spaced photon energies are aligned to provide spectra from single pixels and irregular-sized regions. The spectrum obtained at the NSLS was done using a point spectra method where the 50nm beamspot stays in one position on the sample and the grating is moved continuously from 280-310 eV. There are advantages to both methods but the difference we see in the sigma section of the spectra maybe due to the fact that the dwell time in point spectra are much longer than in spectroscopic image sequences. Otherwise, the overall spectra are nearly identical, showing the graphite  $\pi^*$  peak at 285.4eV and the  $\sigma^*$ peak at 291.8eV. The 288.5eV peak can be assigned to both C-C structure or a C-O functional group. If, in fact, this peak is due to a distortion of the graphite structure, than it may be possible to use carbon XANES to predict variations in nanotubes such as single walled versus multiple walled or metallic versus semi-conducting tubes.

R. Goswami *et al.*, "Diamond synthesis by high-velocity thermal spray: The laboratory analogue of a meteorite impact", J. Mater. Res., 15, 25-28 (2000).



**Figure 1**. Solid line carbon XANES from the X1A beamline, NSLS, dotted line carbon XANES from beamline 7.0.1.1 at the ALS